SYNTHESIS OF CELLULOSE ESTERS CONTAINING ORGANIC PHOSPHORUS

BY

BERNARD RICHARD BLUESTEIN

B.S., University of Pennsylvania, 1946
M.S., University of Illinois, 1947

AN ABSTRACT OF A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN CHEMISTRY
IN THE GRADUATE COLLEGE OF THE
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Introduction

This research was undertaken with the purpose of synthesizing cellulose esters which contain phosphorus linked to the cellulose through a carbon-phosphorus bond without causing extensive degradation or cross-linking of the cellulose. These cellulose derivatives were tested for their resistance to burning.

The industrial flameproofing methods now in use are those which involve the impregnation of the woven cotton fabrics (1). Inorganic phosphates are the main ingredients used in these treatments.

Previous attempts to flameproof cellulose by the direct action of phosphoric acid or phosphorus oxychloride on cellulose fibers have been successful, but the products thus formed were degraded and easily hydrolyzed.

Discussion Of Results

Cellulose acetates containing chlorine as chloroacetyl groups were prepared by causing chloroacetyl chloride in a mixture of pyridine and xylene to react with cellulose acetate which contained some free hydroxyl groups. It was found that the chloroacetylation could be carried almost to completion and with little degradation occurring. This method was similar to that of Barnett (2), but he stated that the reaction was difficult to control and that charred products were formed very easily.

Cellulose acetates containing chlorine were prepared also by the use of chloroacetic anhydride and concentrated sulfuric acid as a catalyst by using Rudy's procedure (3), but the cellulose derivatives were extensively degraded. The use of dil-

uents to retard the degradation also caused a retardation of the chloroacetylation.

Cellulose acetates containing phosphorus as diethyl phosphonoacetyl groups were prepared by an Arbuzov reaction (4) of the cellulose acetate chloroacetates with triethyl phosphite. The ethyl chloride gas evolved during the reaction was collected in a graduated cylinder over water and the amount of gas collected was plotted against the time of the reaction. The curve was similar in appearance to that of a known Arbuzov reaction, triethyl phosphite with ethyl chloroacetate (5), which was also plotted.

The cellulose derivatives, which contained as little as 1.10 per cent phosphorus, were flameproof. The color of the derivatives were the same as their cellulose chloroacetate reactants. Not all of the products were soluble in the usual cellulose solvents.

Diethyl chlorophosphonate, which was prepared from diethyl phosphite and sulfuryl chloride, was caused to react with cellulose acetate containing 2.09 acetyl groups per glucose unit, in the presence of a mixture of chloroform and pyridine, to form a cellulose acetate diethyl phosphate. This product was white, insoluble, and flameproof.

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VITA

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